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“Step Out From the Old to the New”

IS 12473-2 (1988): Chemical Analysis of Hardmetals by Flame Atomic Absorption Spectrometry, Part 2: Determination of Calcium, Potassium, Magnesium and Sodium in Contents from 0 [MTD 25: Powder Metallurgical Materials and Products]



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“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”

Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

**CHEMICAL ANALYSIS OF HARDMETALS
BY FLAME ATOMIC ABSORPTION
SPECTROMETRY**

**PART 2 DETERMINATION OF CALCIUM, POTASSIUM, MAGNESIUM
AND SODIUM IN CONTENTS FROM 0.001 TO 0.02 PERCENT (m/m)**

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Indian Standard

CHEMICAL ANALYSIS OF HARDMETALS BY FLAME ATOMIC ABSORPTION SPECTROMETRY

PART 2 DETERMINATION OF CALCIUM, POTASSIUM, MAGNESIUM AND SODIUM IN CONTENTS FROM 0.001 TO 0.02 PERCENT (*m/m*)

0. FOREWORD

0.1 This Indian Standard (Part 2) was adopted by the Bureau of Indian Standards on 23 September 1988, after the draft finalized by the Powder Metallurgical Materials and Products Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 Chemical analysis of hardmetals by flame atomic absorption spectrometric methods is covered in the following parts:

	<i>Percent Range of Each Element (<i>m/m</i>)</i>
Part 1 General requirements	—
Part 2 Determination of calcium, potassium, magnesium and sodium	0.001-0.02
Part 3 Determination of cobalt, iron, manganese and nickel	0.01-0.5
Part 4 Determination of molybdenum, titanium and vanadium	0.01-0.5

Part 5 Determination of cobalt, 0.5-2.0
iron, manganese, molybdenum,
nickel, titanium and vanadium

Part 6 Determination of chromium 0.01 - 2.0

NOTE — The method for determination of chromium also permits determination of iron, nickel and manganese within the range 0.01 to 2 percent.

0.3 In the preparation of this standard, assistance has been derived from ISO 7627/2-1983 'Hardmetals — Chemical analysis by flame atomic absorption spectrometry, Part 2: Determination of calcium, potassium, magnesium and sodium in contents from 0.001 to 0.02 percent (*m/m*),' issued by the International Organization for Standardization (ISO).

0.4 In reporting the result of the test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard (Part 2) specifies the method to be used for the determination of calcium, potassium, magnesium and sodium contents in hardmetals within the range 0.001 to 0.02 percent (*m/m*) by flame atomic absorption spectrometry.

1.2 General requirements concerning the field of application, principle, interfering elements, apparatus, sampling and test report are given in Part 1 of this standard.

NOTE — In low concentrations, the determination of these elements is very critical. Every care should be taken to avoid contamination from atmosphere and reagents.

2. REAGENTS

2.1 During the analysis, only reagents of analytical grade, and distilled water or water of equivalent purity shall be used.

2.1.1 *Hydrofluoric Acid* ($\rho = 1.12$ g/ml)

2.1.2 *Concentrated Nitric Acid* ($\rho = 1.42$ g/ml)

2.1.3 *Ammonium Fluoride* (0.1 g/ml solution)

2.1.4 *Caesium Chloride* (0.01 g/ml solution)

2.1.5 *Standard Caesium Solution* (1 ml = 1 mg of Ca) — Weigh (to the nearest 0.000 1 g) in a beaker

2.497 2 g of calcium carbonate previously dried at 110°C and cooled in a desiccator. Add 10 ml of concentrated hydrochloric acid. When dissolution is complete, transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

2.1.6 Standard Potassium Solution (1 ml = 1 mg of K) — Dry 3.0 g of potassium chloride at 110°C. Take 1.906 8 g of potassium chloride and dissolve in 100 ml of water. Transfer to 1 litre volumetric flask, dilute with water up to the mark and mix.

2.1.7 Standard Magnesium Solution (1 ml = 1 mg of Mg) — Weigh 1.000 0 g of pure magnesium metal (purity 99.95 percent) in a 250 ml beaker. Add 25 ml dilute hydrochloric acid (1 : 1). Boil gently to dissolve the metal. Add 50 ml of dilute hydrochloric acid. Transfer to 1 litre volumetric flask, dilute with water up to the mark and mix. Store in a polyethylene bottle.

2.1.8 Standard Sodium Solution (1 ml = 1 mg of Na) — Weigh 3.089 1 g of anhydrous sodium sulphate previously dried at 110°C and cooled in a desiccator. Place it in a 250 ml beaker and add 10 ml of concentrated hydrochloric acid. Transfer to 1 litre volumetric flask, dilute with water up to the mark and mix. Store in a polyethylene bottle.

3. PROCEDURE

3.1 Test Portion — Weigh, to the nearest 0.001 g, approximately 1 g of the test sample. Transfer it to a 100 ml polytetrafluoroethylene beaker. Cover the beaker.

3.2 Dissolution of the Test Portion — Add 10 ml of water, 5 ml of hydrofluoric acid and then 5 ml of concentrated nitric acid, drop by drop, to the beaker containing the test portion and heat gently until the test portion is completely dissolved. Add 10 ml of caesium chloride solution and 10 ml of ammonium fluoride solution. Then transfer the solution totally

to a 100 ml polyethylene volumetric flask and dilute to the mark and mix.

3.3 Dilution Volume — Prepare the relevant dilution volume for analysis according to Table 1 as follows.

3.3.1 Dilution Volume (100 ml = 1 g of test sample) — Use the solution 3.2.

3.3.2 Dilution Volume (1 000 ml = 1 g of test sample) — The concentration of the solution may be reduced by a factor of 10 for instruments of higher sensitivity by transferring 10 ml of the solution in 3.2 to a 100 ml polyethylene volumetric flask. Add 10 ml of caesium chloride solution. Add 10 ml of ammonium fluoride solution and dilute to the mark.

3.4 Preparation of Calibration and Blank Solutions

3.4.1 Prepare at least six solutions according to 3.2 with a matrix composition as similar as possible to the test portion to be analyzed but without making up to volume. Then add increasing volumes of properly diluted standard solutions (2.1.5 to 2.1.8) of the elements to be determined according to the concentration ranges to be covered. Make up to 100 ml and mix.

3.4.2 Also prepare calibration solutions with a diluted matrix in accordance with 3.3.2 for dilution volumes of 1 000 ml, if necessary.

3.4.3 Prepare at least two blank solutions (see 3.4.1) without the addition of relevant element to be determined.

3.5 Adjustment of the Atomic Absorption Spectrometer — Ignite the flame. Optimize the response of the instrument at the wavelength given in Table 1 for the element being determined. Preheat the burner for about 5 minutes and then adjust the fuel and correct the burner to obtain maximum absorption while aspirating a calibration solution. Make sure that the absorbance reading is not drifting. Aspirate water and set the initial reading to zero absorbance.

TABLE 1 INSTRUMENTAL PARAMETERS AND CHARACTERISTICS OF
CALIBRATION FUNCTIONS
(Clauses 3.3 and 3.5)

ELEMENT	DILUTION VOLUME (V) FOR 1 g TEST PORTION*	OXIDANT	WAVE LENGTH	RECIPROCAL SENSITIVITY FOR 1 PERCENT ABSORBANCE*	LINEAR RANGE*	NOTES
	ml		nm	µg/ml	Percent	
(1)	(2)	(3)	(4)	(5)	(6)	(7)
Ce	100	N ₂ O	422.7	0.03	0.00 1 to 0.02	1
K	100	Air	769.9	0.04	0.000 5 to 0.02	
Mg	100	N ₂ O	285.2	0.02	0.000 3 to 0.04	
Na	100	Air	589.3	0.02	0.000 5 to 0.02	2

NOTE 1 — Sensitivity is greatly dependent on matrix composition.

NOTE 2 — Use the doublet.

*Guidelines for information only.

3.6 Atomic Absorbance Measurements

3.6.1 Aspirate first the blank solution and then the calibration and test solutions consecutively and record the readings. Aspirate water between each solution. Make at least two measurements for each solution. Solids which build up on the burner slit must be removed, otherwise it will cause a decrease of sensitivity.

3.6.2 Prepare a calibration curve by plotting the obtained absorbance values of the calibration solutions corrected for the blank against the concentration, in mg per litre, of the element.

3.6.3 Convert the absorbance values of the test solutions corrected for the blank of mg of the element per litre by means of the calibration curve.

4. TEST RESULTS

4.1 Calculation — The element content, expressed as a percentage by mass, is given by the formula:

$$\frac{C \times V}{10^4 \times m}$$

where

C = concentration, in mg per litre, of the element in the test solution;

V = dilution volume, in ml; and

m = mass, in g, of the test portion.

4.2 Permissible Tolerances — The deviations between three independent determinations shall not exceed 0.000 5 percent (absolute value) at the lower limit of determination. It shall not exceed 0.002 percent at the 0.02 percent level.

4.3 Final Result — Report the arithmetical mean of acceptable determinations rounded to 0.001 percent. If the element content is below 0.001 percent or greater than 0.02 percent, report the result as less than 0.001 percent or greater than 0.02 percent, respectively.